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ELEVATED TEMPERATURE PROPERTIES OF SHORT FIBER REINFORCED ALUMI--ETC(U)

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Elevated Temperature Properties of Short Fiber Reinforced Aluminum

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Recently two new reinforcing fibers have been developed, which have the potential for selling ~~at~~ roughly \$22/kg (\$10/lb). They are the very small SiC whiskers produced from rice hulls, an inexpensive by-product of the food processing industry, and graphite fiber mats produced directly from pitch without an intermediate yarn precursor. SiC/2024 Al and G/2024 Al composites with 21 volume percent reinforcement were manufactured by pressure casting ingots with random fiber orientations and then warm extruding the ingots to get a good degree of fiber alignment. For over
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20. Abstract (Continues)

comparison purposes, a unreinforced 2024 Al control was subjected to the same thermomechanical treatment. Tensile properties were evaluated over a temperature range from room temperature to 380°C (720°F). The G/Al had poorer mechanical properties than unreinforced Al because there was excessive fiber breakage during extrusion. SiC/Al had a Young's elastic modulus and a specific elastic modulus which were both 50 percent greater than the values for wrought aluminum at temperatures up to 300°C (570°F). At temperatures less than 200°C (390°F), the SiC reinforced composite had 30 percent greater yield strength and fifteen percent greater tensile strength than the Al control. These SiC/Al data are encouraging because of the low projected cost of the whiskers, the economics associated with manufacturing composites by casting and extrusion, and the ease with which the composite can be cut using conventional steel tools.

CONTENTS

ELEVATED TEMPERATURE PROPERTIES OF SHORT FIBER REINFORCED ALUMINUM

INTRODUCTION

Metal matrix composites made with strong, stiff whiskers have not been used in structural designs because their properties are not good enough to justify the extremely high cost of the whiskers, which run into the hundreds, or even thousands, of dollars per kilogram (1, 2). Recently, two new reinforcing fibers have been developed which have the potential for selling at roughly $22/kg ($10/lb). They are the small SiC whiskers produced from rice hulls, a by-product of the food processing industry, (3) and the graphite fiber mats produced directly from pitch without an intermediate yarn precursor (4). The G fibers are not whiskers, but, because they are discontinuous, from a composites point of view, they are more closely associated with whisker technology than continuous filament technology. The cost of metal matrix composites made with the SiC and G fibers will depend on many factors including the production volume. An estimate of $22/kg ($10/lb) appears to be realistic for structural shapes with 30 volume percent reinforcement provided the composites can be mass produced using inexpensive casting and extrusion methods. If cost can be held to this level, even a modest increase in mechanical properties, over the properties obtainable from wrought metals, will make these composites economically competitive for a wide range of high performance structural applications.

One of the more serious limitations of wrought aluminum is its decrease in strength at relatively low elevated temperatures. Reinforcing Al with continuous filaments of B, BORSIC and G significantly extends the temperature range of the matrix (5, 6, 7). Similarly, reinforcing Al with Al_2O_3 and SiC whiskers has been shown to improve the high temperature properties of the matrix, but to a lesser degree than the continuous filaments (8, 9). Direct comparison between the continuous and discontinuous reinforcement can be deceptive, however, in that axial tensile tests of continuous filament composites are almost always conducted with cool grips and continuous filaments spanning the distance between the grips. As such, the

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continuous filament high temperature test really measures the fiber bundle strength, which may, or may not, relate to the strength of the composite part, depending on the temperature distribution in the component (10). This is quite different from the hot test of a short fiber composite where the entire filament is in the hot zone. Since the strength of a short fiber composite depends on many things, such as the quality of the fiber/matrix bond, the fiber shape, the residual stress, etc., elevated temperature properties must be evaluated for each new reinforcing material.

The objectives of this research are twofold. First, to determine if the SiC whiskers produced from rice hulls and the G fibers produced directly from pitch can be successfully manufactured into Al matrix composites by pressure casting ingots with a random fiber orientation and then warm extruding the ingots to produce a good degree of fiber alignment, and second, to evaluate the short time elevated temperature strengths and stiffnesses of the SiC/2024 Al and the G/2024 Al composites.

MATERIALS AND TESTING PROCEDURE

The SiC whiskers were manufactured by Silag, Inc., a joint venture of Exxon Corp. (80 percent) and Commercial Technology, Inc. (20 percent). One of the reasons for the potentially low cost of these whiskers is that they are made from rice hulls, which are available from food processors at a minimal charge. Figure 1 shows the single crystal SiC prior to composite manufacture. It is estimated that 65 percent of the SiC is in the form of whiskers, which are mostly hexagonal in cross section but, for simplicity, are approximated by equivalent diameters of 0.2 to 0.5 μm (8 to 20 μinches) and have length to diameter ratios of roughly 80. The remainder of the material is in the form of small SiC particles.

The G fibers were in the form of a VMA pitch based mat produced by Union Carbide. Since the intent was to use low cost materials and methods, no surface treatment, such as the chemical vapor deposition of a thin coating material, was applied to the G fibers to promote fiber/matrix bonding.

SiC whiskers, along with the small SiC particles, and G fibers were fabricated into composites by Divecha Associates using a proprietary process which basically consisted of liquid metal pressure infiltrating the fiber reinforcements with 2024 Al at 670 ± 20°C (1240 ± 36°F). The resulting 25 mm (1.00 inch) diameter by 32 mm (1.25 inch) long ingots had a random fiber orientation before they were extruded

into 6.4 mm (0.25 inch) diameter rods at a temperature of 480 ± 50°C (900 ± 90°F). The 16:1 area reduction during extrusion was designed to put the material in a more usable dimensional form and to give the fibers a good degree of alignment in the axial direction. Extrusion was accomplished at 50 mm/min (2.0 inch/min using a mirror finished carbide die with a 3.2 mm (0.125 inch) radius at the entry and a 1.6 mm (0.062 inch) straight section blended in (11). A 9.5 mm (0.375 inch) thick disc was placed in front of each billet to clad the composite and to prevent the die from being abraded by the fibers during extrusion. For comparison purposes, 2024 Al control billets were cast and extruded by the same process and at the same temperatures except that no reinforcement was used. Densities of the unextruded ingots and the extruded rods are given in Table 1, along with the volume percent of reinforcement. The amounts of reinforcement were determined by the rule of mixtures assuming a pitch based G density (4) of 2.00 g/cm^3 and a SiC density (12, 13) of 3.197 g/cm^3. The 2024 Al was chosen as the matrix because it has a high strength when heat treated. In this study, all materials were tested in the "as fabricated" condition with no special heat treatment other than that associated with the warm extrusion and the strain gage bonding, which will be discussed later.

Two SiC/Al and two G/Al specimens with a single reduction gage section were tensile tested at room temperature. Elastic moduli and yield strengths appeared to be valid but the fracture data were not used because failure occurred at the change in diameter. To solve this problem, 25.4 mm (1.00 inch) long specimens with a double reduction gage section of the design shown in Fig. 2 were used for all of the remaining tests. The main data format was a plot of load versus longitudinal strain, where strains were measured with two BLH FSM 06-35 strain gages bonded to opposite sides of the specimen with MicroMeasurements M-Bond 610 epoxy-phenolic adhesive. Both gages and adhesive were rated for limited service to 370°C (700°F). By electrically connecting the gages to diametrically opposite sides of a Wheatstone bridge, bending strains due to specimen misalignment cancel and the elastic portions of the load-strain plots were linear. This allowed an accurate measurement of the Young's elastic moduli. Some problems were encountered in applying the relatively stiff polyimide backed strain gages to the 3.56 mm (0.140 inch) diameter specimens mainly because the high temperature adhesive required heat curing with contact pressure exerted on the gage. The bonding procedure was to clamp the specimen in a fixture, apply one gage and partially cure the adhesive at 149°C (300°F) for 30 minutes while maintaining contact pressure with a spring loaded cloth strap. The specimen was slowly cooled, rotated

180 degrees on the bonding fixture, and the other gage applied. A second adhesive curing cycle consisted of 149°C (300°F) for two hours with spring pressure applied to the second gage. Despite care in applying the gages, some failures were encountered. As a backup to the load-strain data, a separate load-time plot was made during most of the tests.

Temperatures were measured with 0.15 mm (0.006 inch) diameter Type K thermocouples staked into 0.35 mm (0.014 inch) diameter holes drilled in the specimen head, as shown in Fig. 2. Positive and negative wires were inserted into separate holes to form an intrinsic couple, care was taken to assure that the only conducting path between the wires was through the specimen, and a drop of barrier coat was applied to insulate the area from direct heat input. Thermocouple output was displayed on the dual pen X-Y recorder used for the load-strain data.

Specimens were heated with a 3 zone, high intensity, tungsten lamp furnace having a 57 mm (2.25 inch) long heating zone. The center lamp was disconnected to prevent excessive thermal input to the non-conducting strain gage backing. Dimensions were such that the grips were heated with the top and bottom lamps and the heat flowed into the sample across the specimen/grip interfaces. A 10 kg (22 lb) tensile load was maintained during heating to assure good thermal conductivity between the specimen and the grips. Electrical current to the furnace was determined by two process controllers, each adjusted to provide rapid heat-up with no overshoot and regulated by its own thermocouple attached to the appropriate grip. Preliminary heating tests were conducted with thermocouples embedded in holes drilled in the gage section and in the specimen head. This sample, which was not tensile tested, verified that the heat-up times at the gage section were only a few seconds longer than at the head and that the final temperatures at the two locations were the same.

Elevated temperature tests were conducted by heating the specimens for 90 seconds, with approximately 30 seconds required for heat-up and the remaining 60 seconds used for an elevated temperature soak. Specimens were tensile tested using an Instron at a crosshead speed of 5.1 mm/min (0.20 inches/min). For a rigid machine, this would correspond to a strain rate of 5×10^{-3} sec^{-1}.

RESULTS

Young's elastic modulus was measured at room temperature on most of the SiC/Al and Al control specimens after they

had been installed on the Instron and the furnace positioned for elevated temperature testing. The procedure was to apply a series of increasing loads, stop the Instron, and manually record the strain gage output after 10 seconds. In no case did the applied stress exceed 70 MPa (10 ksi), which is well below the proportional limit. Room temperature elastic moduli were calculated from straight lines drawn through the manually recorded data and the results are given in Table II. The mean elastic modulus for the SiC/Al was 120 GPa (17.3×10^6 psi) with a 1 σ deviation of 12 GPa (1.7×10^6 psi) and for the Al control was 77.3 GPa (11.2×10^6 psi) with a 1 σ deviation of 6.6 GPa (1.0×10^6 psi). The handbook modulus value for wrought 2024 Al is 73 GPa (10.6×10^6 psi) (14).

Figure 3 and Table II give the temperature dependence of the Young's elastic modulus as calculated from the linear region of the stress-strain curves. In these calculations, data provided with the strain gages were used to correct for the temperature dependence of the gage factor. Straight lines with negative slopes have been drawn through the data in Fig. 3 in agreement with the results reported in the literature for wrought aluminum (15). The tabulated room temperature moduli and the plotted elevated temperature values show that the SiC reinforced composite is 50 percent stiffer than unreinforced wrought aluminum and that this increase in stiffness is maintained to at least 300°C (570°F). G/Al, on the other hand, is slightly less stiff than unreinforced aluminum.

A similar, but, in some ways more significant, comparison can be made based on the specific elastic modulus, which is defined as the Young's elastic modulus divided by the mass per unit volume. Figure 4 gives the temperature dependence of the specific modulus in units of $Mm^2 \cdot s^{-2}$ ($in^2 \cdot sec^{-2}$). These rather unusual dimensions for specific modulus occur because weight and mass are not equivalent. Since the SiC/Al composite is only 3 percent more dense than aluminum, it has a 45 to 50 percent greater stiffness than wrought aluminum, whether the comparison is based on Young's modulus or specific modulus. G/Al is 6 percent less dense than unreinforced aluminum, which makes the specific modulus of the G/Al composite the same as that for the Al control.

Temperature dependence of the 0.2 percent offset yield strength is tabluated in Table II and plotted in Fig. 5. Yield strengths were determined by two methods. They were measured from the load-strain curves and calculated from the load-time curves using known values of gage length, crosshead and chart speeds. Comparison of the results for 12 specimens

where both types of data are available show a mean difference in yield strength for the same sample of 5 percent and that the load-strain curve gives the higher yield strength for 6 out of the 12 specimens. Strength values based on load-strain curves were used in all cases where there were suitable data. For samples that experienced early gage failure, yield strengths calculated from load-time plots were used. These data are denoted by superscripts in Table II.

At temperatures less than 200°C (390°F), the SiC reinforced aluminum had a 0.2 percent offset yield strength of 293 MPa (42.5 ksi). This is 30 percent greater than the value for the wrought 2024 Al control which had the same thermomechanical history. The yield strength of the G/Al composite over the same temperature range was 25 percent less than that recorded for the unreinforced Al. For temperatures greater than 200°C (390°F), the yield strengths of all three materials are temperature dependent. There is some scatter, but, at a given strength level, the SiC/Al composite appears to be capable of withstanding roughly 20°C (36°F) higher temperatures than G/Al or wrought Al.

Figure 6 and Table II give the temperature dependence of the engineering ultimate tensile strength for SiC/Al and G/Al composites and the Al control. When the temperatures are less than 200°C (390°F), the SiC/Al is 15 percent stronger than the unreinforced Al but the G/Al is as much as 30 percent weaker. Between 250° and 300°C (480° and 570°F), the SiC whiskers appear to bring about roughly a 15°C (27°F) increase in temperature range for a given short time strength requirement.

Plastic fracture strains, defined as the total fracture strains minus the elastic component of the fracture strains at the fracture loads, are given in Table II and Fig. 7 as a function of temperature. At temperatures less than 240°C (460°F), both composites failed with relatively little plastic flow and no necking. Fracture strains for these specimens were calculated from load-time records. Composite samples tested at higher temperatures and all of the aluminum control samples displayed substantial plastic flow and necked prior to fracture. For the ductile specimens, fracture strains were defined as the true local strains at the neck. These strains were calculated from the initial diameter, D_i, and the minimum diameter at the neck after fracture, D_f, using the equation (16):

$$\varepsilon_p = 2 \ln(D_i/D_f) \qquad (1)$$

Two methods for measuring strains were necessary because, at low plastic flow, ϵ_p values obtained using Eq. 1 were very sensitive to small inaccuracies in measuring D_i and D_f. On the other hand, the load-time curves could not be used for large fracture strains because chart speeds had been chosen to amplify the early portion of the records at the expense of having the latter portion of the curves fall off the scale.

DISCUSSION

Continuous graphite filaments have been found to be effective in increasing the axial tensile properties of Al, but the fiber and manufacturing costs for these composites are high (17, 18, 19). It was expected that discontinuous G mat reinforced Al composites manufactured by low cost casting and extrusion methods would also show an improvement in axial tensile properties, but the amount of improvement would be less than for continuous filaments. Data in Figs. 5 through 7 show that the short fiber G/Al actually had a lower, not a higher, yield strength, tensile strength and ductility than the unreinforced Al control at temperatures less than 200°C (390°F), and that, compared to the control, there was no improvement in strength at higher temperatures. The reason for the poor properties is shown in Fig. 8, a photomicrograph of a polished cross section of G/Al looking normal to the extrusion direction, and Fig. 9, a photograph of G fibers which had been leached from the extruded composite. The figures reveal that the fibers were well aligned in the axial direction but that there had been excessive fiber breakage during extrusion. Numerous studies have shown that, up to a point, composite strength increases with increasing fiber length to diameter ratio. For the extruded G/Al, the typical length to diameter ratio was less than 3. This is way too short to obtain significant fiber strengthening and the negative effects of voids, fiber/matrix debond and internal stress concentrators combine to reduce the composite strength.

The SiC reinforced Al composite was 50 percent stiffer than the unreinforced matrix material, where stiffness values were expressed as either the Young's elastic modulus, as shown in Fig. 3, or the specific elastic modulus, as shown in Fig. 4. Moreover, stiffness decreased only 5 percent as the temperature was increased from room temperature to 300°C (570°F). To the design engineer, there are two reasons why elastic modulus is one of the most important material properties. First, many high performance structures are "buckling critical", and, for a given cross sectional geometry, buckling loads increase linearly with modulus. Second, stiff structures have high natural frequencies and a high natural frequency is often needed if disastrous resonances, acoustic fatigue and panel flutter are to be avoided.

The room temperature yield and tensile strengths of certain Al alloys, when properly heat treated, are greater than those reported for the SiC/Al composite. This higher strength is generally not usable for structures subjected to prolonged elevated temperature exposure, however, because the material overages and softens with time at temperature. Valid comparisons between the reinforced and unreinforced aluminum, particularly at elevated temperatures, must be based on the same thermomechanical history and the same strain rate. Compared to the Al control, the SiC/Al composite showed a significant improvement in strength properties at temperatures less than 240°C (460°F) and a slight improvement in strength properties at higher temperatures, but these improvements were accompanied by an order of magnitude decrease in low temperature fracture strain. There is no known data on the fracture toughness of this composite but a plastic fracture strain of only 1.4 percent suggests that the toughness of the composite may be lower than desired.

It had been hoped that the temperature at which the short fiber composites lost their strength would be much higher than wrought aluminum and that this would allow the composites to compete with titanium for certain high temperature applications. For a given strength requirement, SiC whisker reinforcement did result in roughly a 15° to 20°C (27° to 36°F) improvement in the maximum operating temperature compared to unreinforced Al. While the change is in the right direction and could effect some borderline cases, the amount of improvement is small and SiC/Al composites are not expected to be serious competitors to Ti for high temperature applications.

A single whisker of the size used in the SiC/Al composite is too small to be accurately resolved with a light microscope (20). The general distribution of whiskers can be viewed optically, however, as demonstrated in Fig. 10, a polished but unetched section having its normal in the extrusion direction. On a microscopic scale, the composite consists of whisker rich areas, which appear dark, and whisker poor areas, which are light. The presence of whisker poor areas suggests that 21 volume percent whiskers is not the maximum and that increasing the whisker content will bring about even greater improvements in strength and stiffness.

A higher magnification scanning electron micrograph of a whisker rich area on the same cross section is shown in Fig. 11. Here, the surface has been roughened by deep etching and is viewed at 45 degrees to the normal. One reason for showing the micrographs of the polished cross section is to emphasize the extremely small size of the reinforcing whiskers. It is the whisker size which gives the SiC/Al composite one of its more important properties:

it can be cut and machined easily using conventional steel tools. This is a big advantage over many composites.

SUMMARY

The pitch based G/Al composite produced by low cost casting and extrusion methods had poorer mechanical properties than an unreinforced Al control due to extensive fiber breakage which occurred during extrusion.

A SiC/Al composite was produced using the same casting and extrusion methods, but with SiC whiskers manufactured from rice hulls as the reinforcing filaments. The composite was 50 percent stiffer than the aluminum control at temperatures up to 300°C (570°F) and had higher yield and tensile strengths. These findings are encouraging because of the low projected cost of the SiC whiskers, the economics associated with manufacturing composites by pressure casting and extrusion, and the ease with which the composite can be machined using steel tools.

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TABLE I. DENSITY AND PERCENT REINFORCEMENT

Material	Ingot Density* (g/cm^3)	Rod Density* (g/cm^3)	Reinforcement (Vol. %)
2024 Al Control	2.76	2.758	-
G/2024 Al	2.58	2.606	21.0**
SiC/2024 Al	2.83	2.858	21.0

*1 g/cm^3 = 0.03613 $lb/inch^3$

**grid interaction method gave 20.1% with a 1σ deviation of 3.0%

TABLE II. TENSILE PROPERTIES OF SiC/Al AND G/Al COMPOSITES AND Al CONTROL

Material	Test Temp. (°C)	Room Temp. Elastic Modulus (GPa)	Properties at Test Temperature: Elastic Modulus (GPa)	Yield Strength (MPa)	Ult. Strength (MPa)	Fract. Strain (%)
SiC/Al	24	-	110.0	269.4	a	a
SiC/Al	25	109.8	106.0	300.9	437.5	0.9[b]
SiC/Al	26	-	111.1	280.7	a	a
SiC/Al	26	120.6	114.6	288.1	415.8	1.8[b]
SiC/Al	139	115.1	105.1	294.8	409.8	1.5[b]
SiC/Al	182	141.1	141.0	296.1	383.9	1.7[b]
SiC/Al	193	-	-	327.6[b]	396.9	1.3[b]
SiC/Al	231	135.8	-	257.5[b]	328.8	2.4[b]
SiC/Al	257	116.0	107.4	177.3	210.9	12.4
SiC/Al	292	105.5	99.4	143.5	167.2	15.9
SiC/Al	312	-	-	133.4[b]	143.5	16.0
SiC/Al	317	119.9	102.5	108.0	120.7	20.0
SiC/Al	382	130.3	-	61.2[b]	71.6	22.5
G/Al	23	-	68.1	167.2	260.3	0.5[b]
G/Al	24	-	78.3	177.8	a	a
G/Al	273	-	70.5	150.0	168.9	2.9[b]
G/Al	299	-	42.5	109.3	124.5	8.5
G/Al	306	-	-	-	110.9	9.8
Al Control	25	-	75.7	220.2	376.2	21.6
Al Control	27	-	75.4	230.8	372.9	26.2
Al Control	137	85.2	76.2	214.1	310.7	22.2
Al Control	205	80.6	71.5	206.7	296.7	20.1
Al Control	232	81.7	80.1	187.2	265.8	36.0
Al Control	239	64.6	61.2	159.3	221.2	33.2
Al Control	285	78.1	63.3	126.0	136.6	123.9

a. Preliminary specimen with single reduction gage section and nominal 0.8 mm (0.03 inch) shoulder radius. Failure occurred at change of section.

b. Calculated from load-time data.

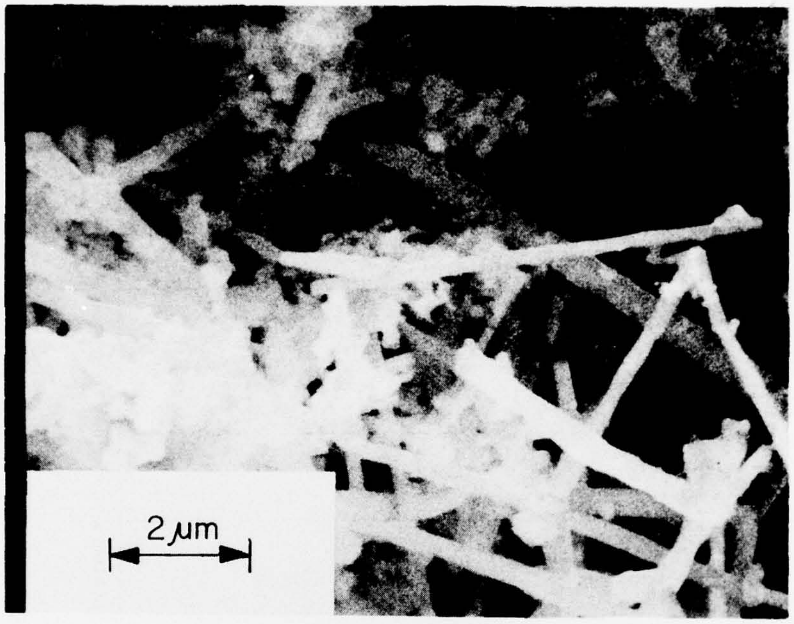

Fig. 1 - Low cost SiC produced from rice hulls. It is estimated that 65 percent of the SiC is in the form of whiskers and 35 percent is in the form of small particles.

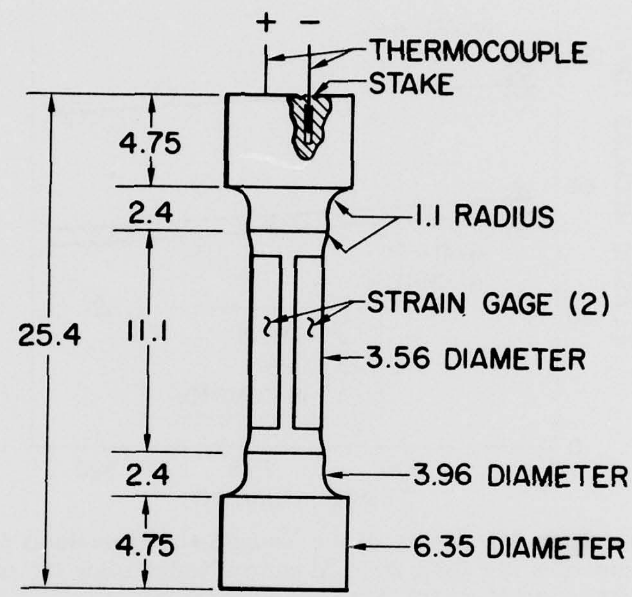

Fig. 2 — Schematic of the tensile specimen showing the double reduction gage section, the location of the strain gages, and the method of attaching the thermocouple leads. Dimensions are in mm (1 mm = 0.0394 inches).

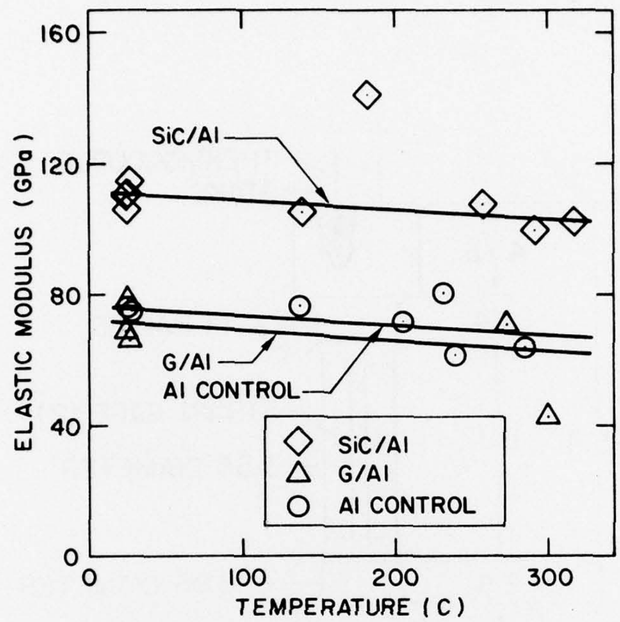

Fig. 3 - Temperature dependence of the Young's elastic modulus for SiC/Al and G/Al composites and for a 2024 Al control tested using the same procedures. (1 GPa = 0.145 × 10^6 psi)

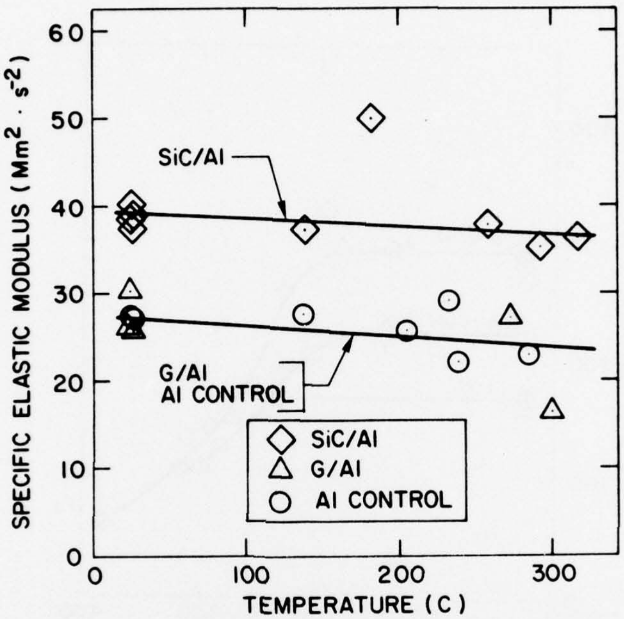

Fig. 4 - Temperature dependence of the specific elastic modulus for SiC/Al and G/Al composites and for a 2024 Al control tested using the same procedures.

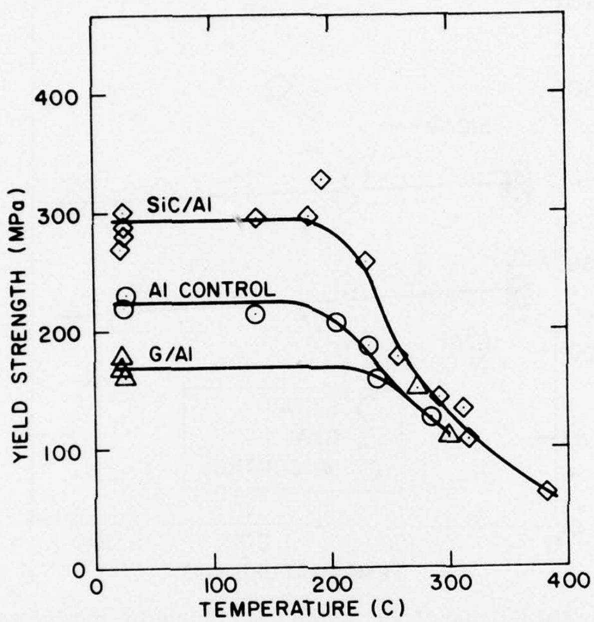

Fig. 5 - The 0.2 percent offset yield strength versus temperature for SiC/Al and G/Al composites and for the 2024 Al control. (1 MPa = 0.145 ksi)

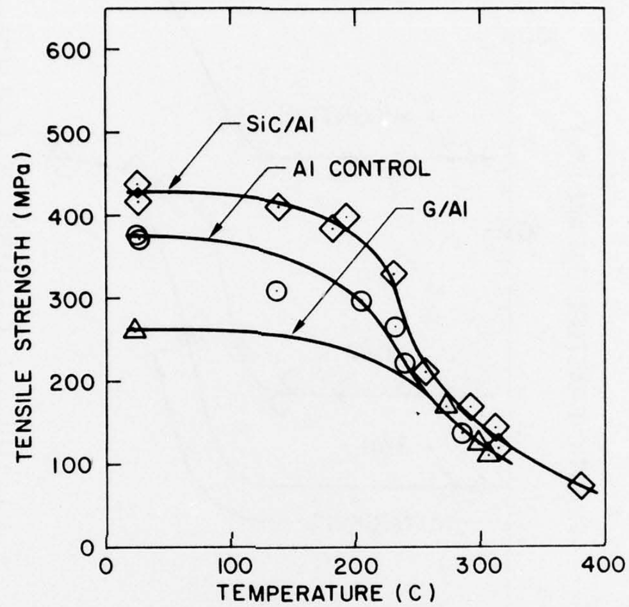

Fig. 6 - Ultimate tensile strength as a function of temperature for SiC/Al and G/Al composites and for the 2024 Al control. (1 MPa = 0.145 ksi)

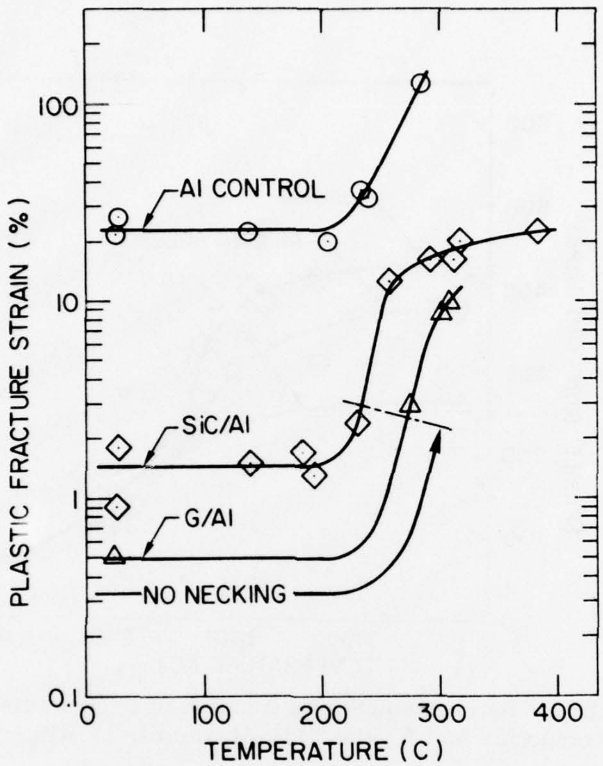

Fig. 7 - Plastic fracture strains plotted on a logarithmic scale versus temperature for the SiC/Al and G/Al composites and for the 2024 Al control.

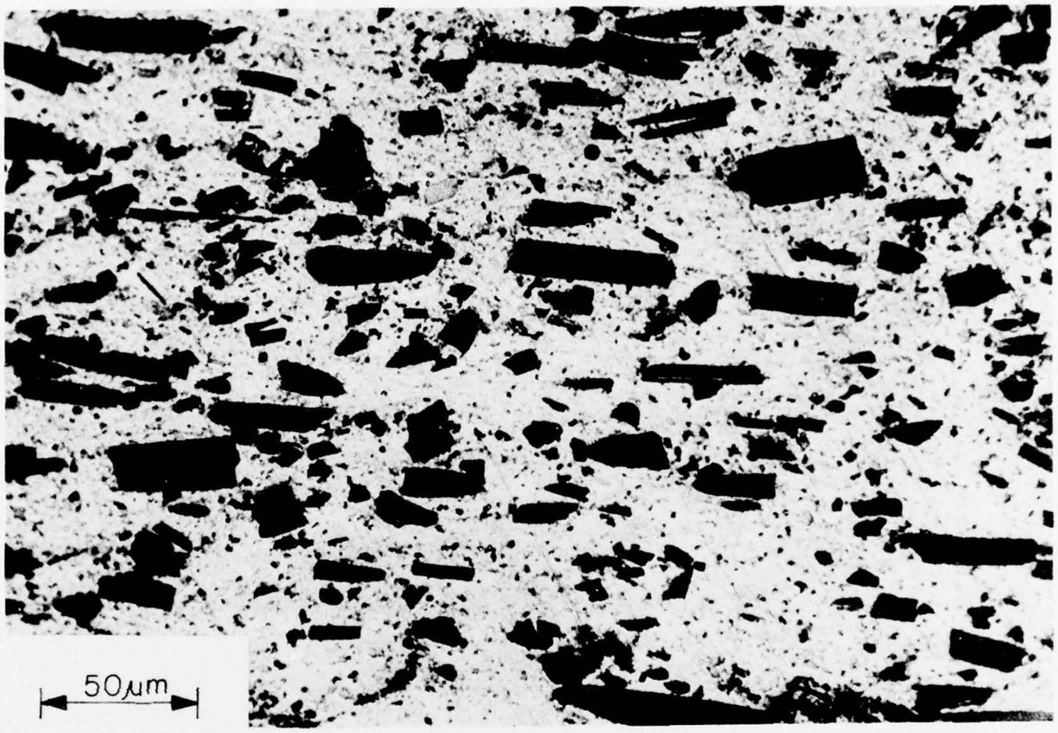

Fig. 8 - Polished cross section of G/Al looking normal to the extrusion direction.

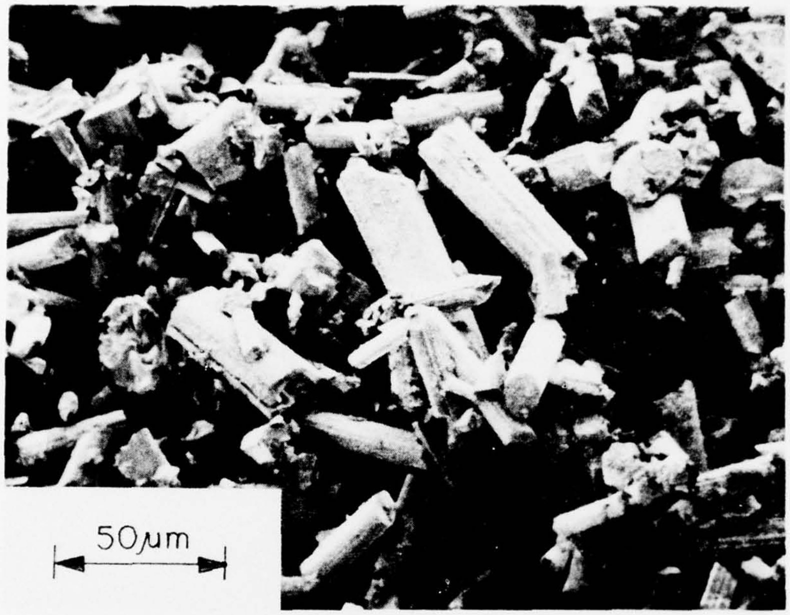

Fig. 9 - Pitch based graphite fiber mat after being leached from the extruded composite.

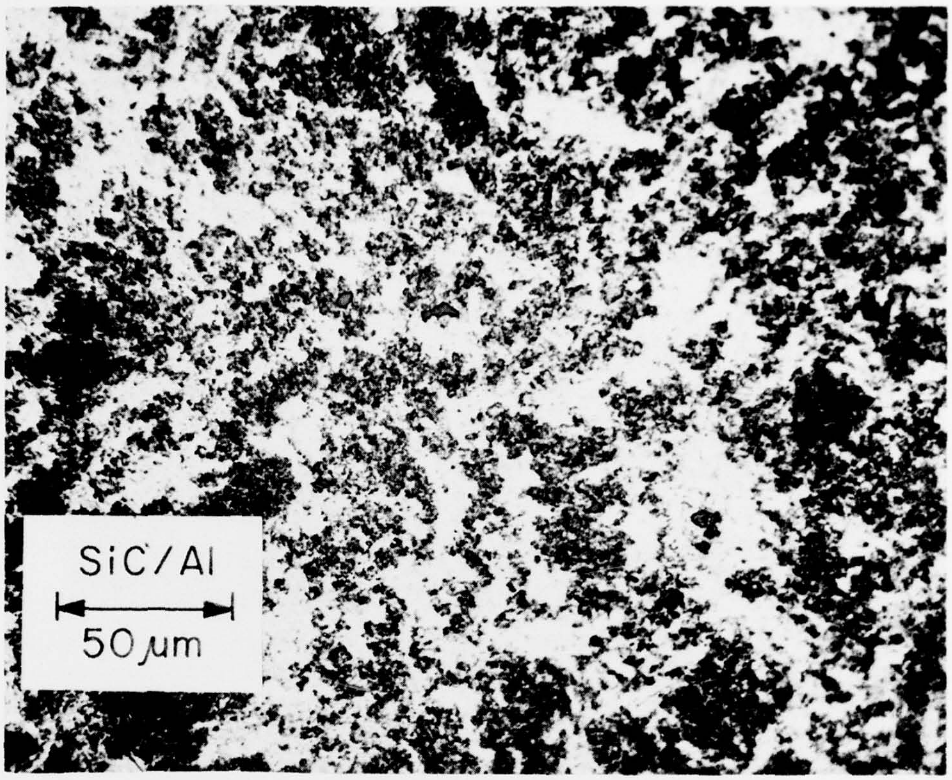

Fig. 10 - Polished but unetched cross section of SiC/Al showing the dark whisker rich areas and the light whisker poor areas. The normal to the surface is in the extrusion direction.

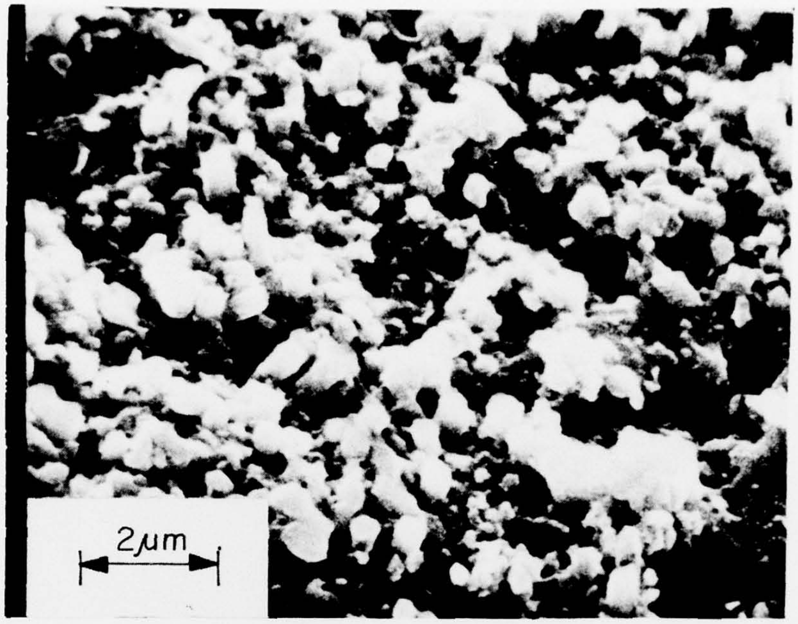

Fig. 11 - High magnification photomicrograph of a whisker rich area on the SiC/Al surface shown in Fig. 10 . View is at 45 degrees to the polished and etched surface.